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VAPOR-DEPOSITED TUNGSTEN

by

Direct Conversion Project Staff

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Lewis Research Center
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TOPICAL REPORT
VAPOR-DEPOSITED TUNGSTEN

by

Direct Conversion Project Staff

Sponsored by
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Lewis Research Center

Technical Management
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J. W. R. Creagh

General Atomic Division of General Dynamics Corporation
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TUNGSTEN DEPOSITION

Chemical vapor deposition of tungsten is basically the deposition of tungsten metal onto a substrate surface by decomposing a volatile tungsten compound either by pyrolysis or by thermochemical reduction. A wide selection of deposition conditions for the forming of tungsten is possible, using a number of thermochemical reactions. These reactions may be generally categorized as follows:

1. Pyrolysis of organo-metallic tungsten compounds, such as tungsten hexacarbonyl, $W(CO)_6 \rightleftharpoons W + 6 CO$;
2. Thermochemical reduction of tungsten halides with a gaseous reducing agent, $WX_6 + 3H_2 \rightleftharpoons W + 6 HX$, where X is a halide;
3. Pyrolysis of a tungsten halide, $WX_6 \rightleftharpoons W + 3X_2$, where X is a halide.

These reactions offer acceptable operating temperatures from 250^o to over 2000^oC. The availability of a substantial number of chemical "tools" for tungsten deposition makes the problem of matching the conditions of the tungsten deposition material to those of a given substrate easier than the similar matching problem for other deposition materials.

Although previously used mainly for cladding various substrate materials, chemical vapor deposition is at present used extensively for forming free-standing structures. For this application the hydrogen reduction of WF_6 is the most widely used process, although some laboratories have worked with the reduction of WCl_6 . Using this process, complex shapes can be formed to wall thicknesses greater than 0.250 in. at rates of up to 1 mil/min over mandrels that are cheap and easily machinable. The resulting structure is a sound, dense, crystalline body of high purity. The equipment used to produce this material is relatively cheap and simple. Figure 1 is a schematic diagram of such a system employed at General Atomic.

SELECTION OF VAPOR-DEPOSITED TUNGSTEN

Early in the development of nuclear-heated thermionic diodes at General Atomic, considerable attention was given to the selection and evaluation of candidate fuel-cladding combinations. Table 1 presents a summary of compatibility data obtained for many systems at 1800^oC, the

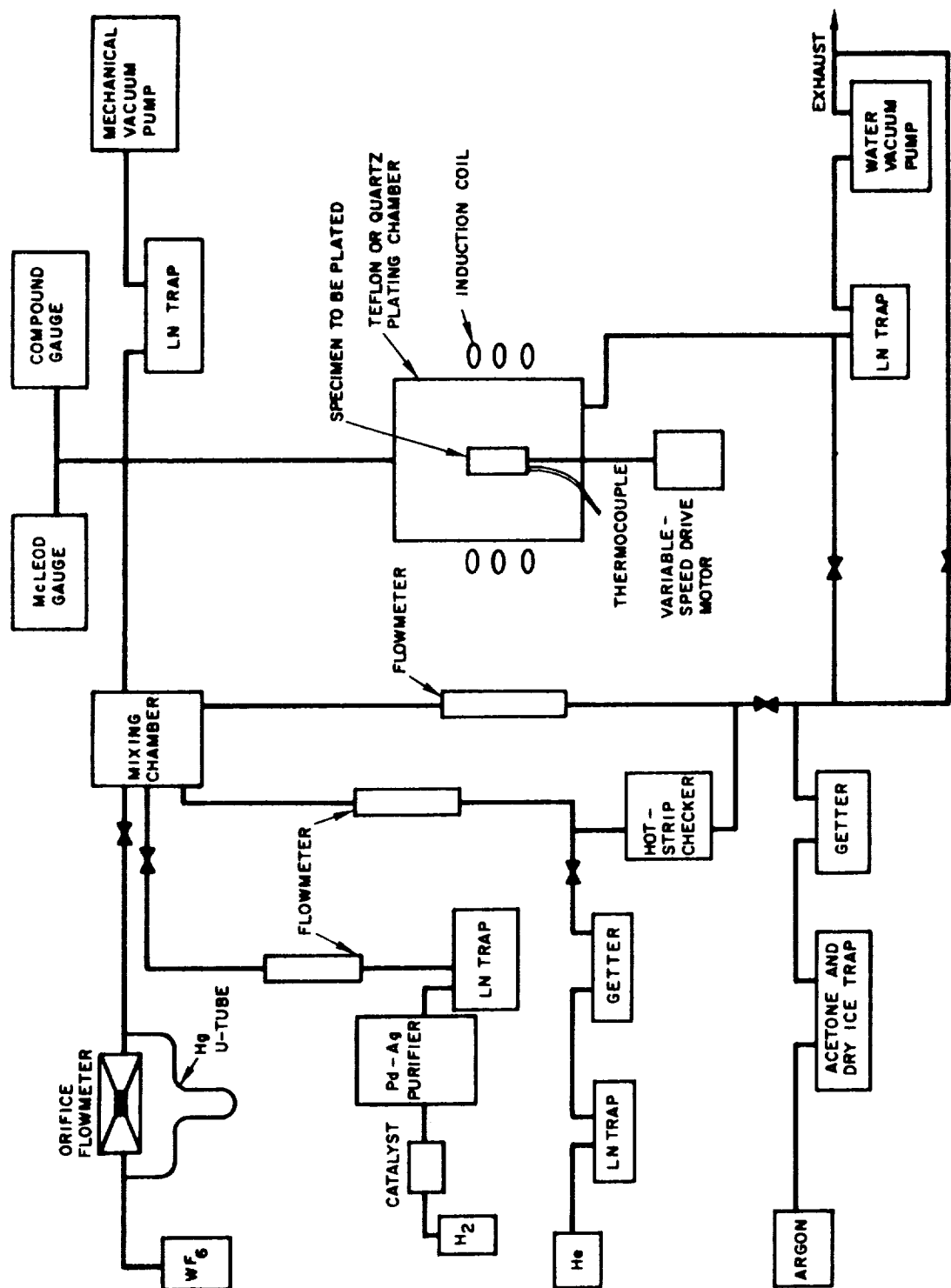


Fig. 1--Schematic diagram of vapor-deposition system at General Atomic

Table 1
SUMMARY OF COMPATIBILITY DATA

Fuel	Temp. (°K)	Refractory Metal								
		Tungsten	Molybdenum	Niobium	Tantalum	Iridium	Rhenium	W-26 Re	W-15 Mo	W-2 Mo
UC	2073	Suitable	Unsuitable ^a	Unsuitable ^a	Unsuitable ^a	Unsuitable ^a	Questionable ^g	Questionable ^b	Unsuitable ^a	Unsuitable ^a
UC ₂	2073	Unsuitable ^c	Unsuitable ^c	Unsuitable ^c	Unsuitable ^c	-----	-----	-----	-----	-----
10 mol-% UC	2073	Suitable	Questionable ^f	Unsuitable ^e	Unsuitable ^e	-----	-----	-----	-----	-----
90 mol-% ZrC	2073	Suitable	-----	-----	-----	-----	-----	-----	-----	Unsuitable ^a
30 mol-% UC	2073	Suitable	-----	-----	-----	-----	-----	-----	-----	Unsuitable ^a
70 mol-% ZrC	2073	Suitable	-----	-----	-----	-----	-----	-----	-----	Unsuitable ^a
90 mol-% UC	2073	Suitable	Unsuitable ^a	Unsuitable ^a	Unsuitable ^a	Unsuitable ^a	-----	-----	-----	Unsuitable ^a
10 mol-% ZrC	2073	Suitable	Unsuitable ^d	Unsuitable ^d	Unsuitable ^d	Unsuitable ^d	-----	-----	-----	-----
UO ₂	2273	Suitable	Unsuitable ^d	Unsuitable ^d	Unsuitable ^d	Unsuitable ^d	-----	-----	-----	-----

^a Liquid-phase formation.

^b Reaction layers or products detected at interfaces.

^c Excessive carbide-layer formation and grain-boundary penetration.

^d Fuel component diffusing through grain boundaries.

^e Uranium and zirconium diffusion through metal at too rapid a rate.

^f Uranium and zirconium penetrated metal slightly.

^g Uranium-bearing ternary intermediate phase formed in cladding.

proposed operating temperature for thermionic systems. It rapidly became evident that whether a carbide or an oxide fuel was used, the only compatible cladding material was tungsten (the only possible exception might be rhenium in the case of UO_2 but this has not yet been firmly established).

Numerous attempts were therefore made to fabricate tungsten emitters from conventional tungsten stock, but all were unsuccessful. The lack of success was due to difficulty of machining and rapid grain growth at the operating temperature. Machining difficulties were associated not with hardness (as electrical discharge machining was used), but rather with an inability to keep the large grains of cast tungsten from "popping" out of sharp corners. Cast tungsten was used because of its greater purity as compared with shapes made from powdered products. Grain growth was a problem, using either type of tungsten stock. This is illustrated in Fig. 2a, in which extruded tungsten tubing is shown after 100 hr at 1800°C . The grains penetrate through the entire wall thickness and the grain boundaries crack readily; they also provide "super highways" for transfer of fuel components to the emitter surface. Figure 2b shows a piece of vapor-deposited tungsten tubing after the same treatment; no grain growth is evident, so the structure is much more stable. This will be discussed in more detail below.

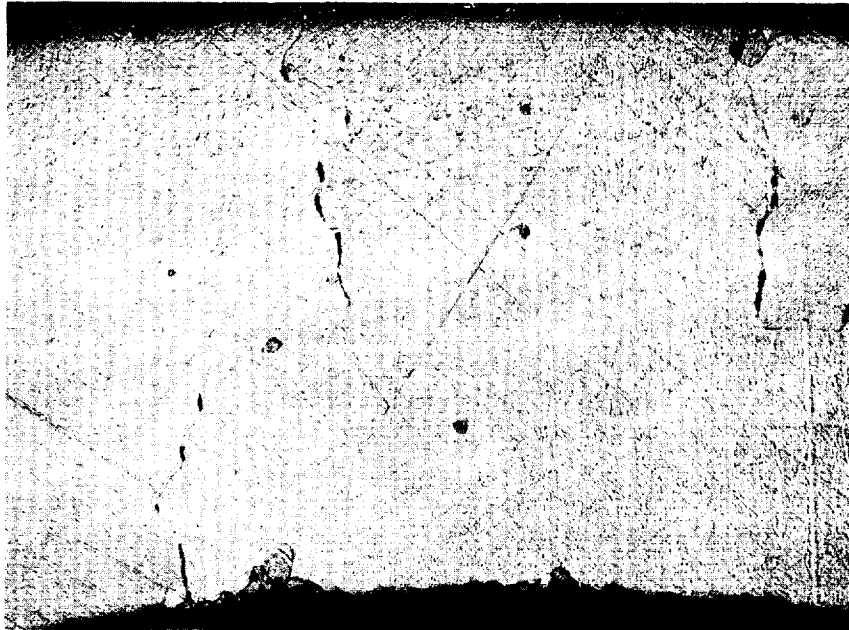
Tungsten-base alloys were investigated, but tungsten-molybdenum alloys proved incompatible with carbide fuels, and tungsten-rhenium alloys have both questionable compatibility and brittleness associated with inhomogeneous structures. It is possible that tungsten-rhenium may prove useful when it is more fully developed.

About the time these problems were being encountered, the use of vapor-deposition techniques to make free-standing shapes was being initiated by several laboratories. Vapor-deposited material was investigated, and it was found that it satisfied thermionic requirements. Some of our experiences with this material and its properties are discussed in the next section.

PROPERTIES OF VAPOR-DEPOSITED TUNGSTEN

Purity

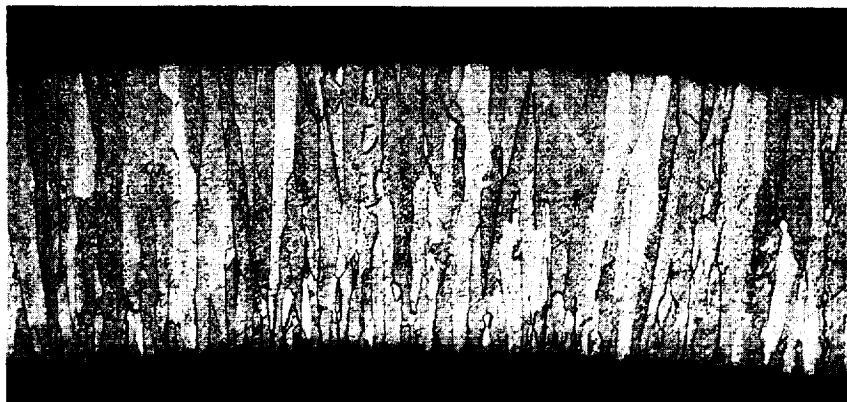
One of the advantages of vapor-deposited tungsten is the extremely high purity obtainable. Table 2 presents a typical chemical analysis of deposited tungsten. Figure 3 illustrates the cleanliness of grain boundaries in this material. One of the important aspects is that not only is the purity exceptionally high but the process is controllable through well-developed chemical engineering approaches, as compared with the irreproducibility



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(75×)

(a) Extruded tubing; note single grains extending through entire tube wall

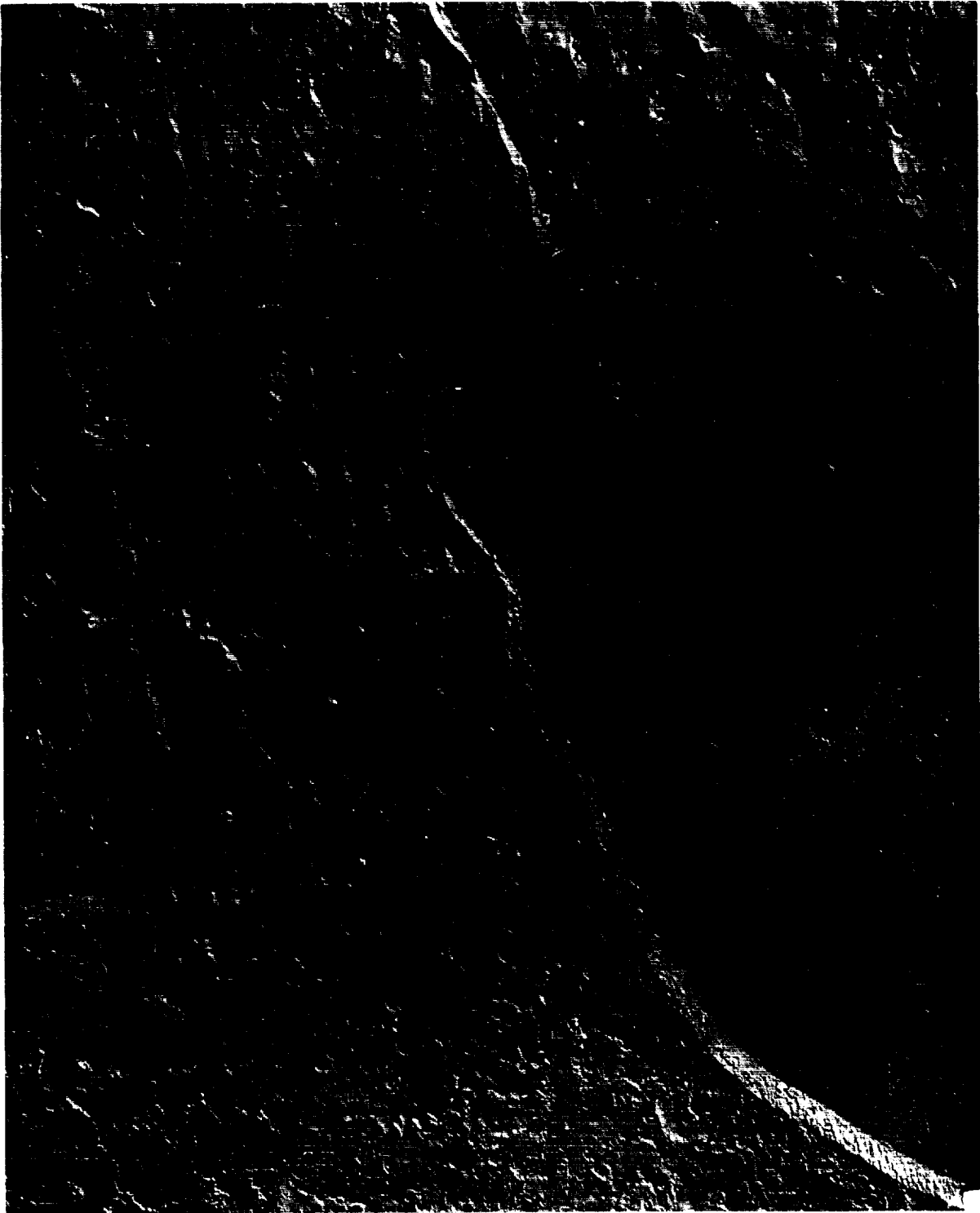


M-8249-1

(100×)

(b) Vapor-deposited tubing; observe retention of small-grained columnar structure, characteristic of material in as-deposited condition

Fig. 2--Grain growth in tungsten annealed at 1800°C for 100 hr



P-3016

(6600×)

Fig. 3--Electron micrograph of grain boundary in vapor-deposited tungsten (note absence of any foreign phase in boundaries)

of wrought material made from stock prepared by conventional extractive metallurgy techniques.

Table 2
TYPICAL PURITY OF VAPOR-DEPOSITED TUNGSTEN

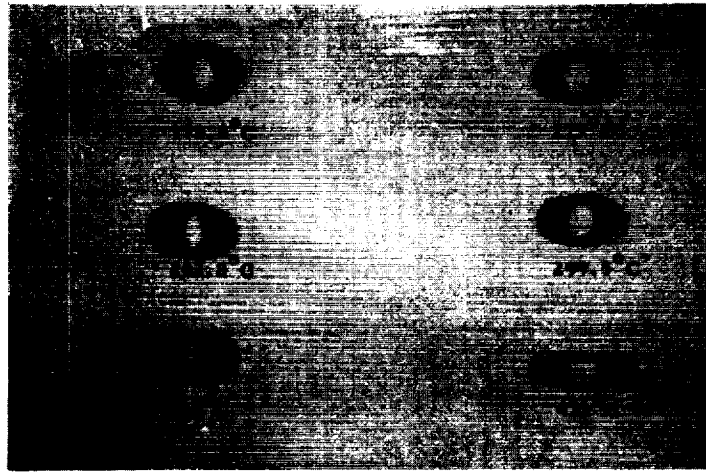
Element	Concentration (ppm)	Element	Concentration (ppm)
O ₂	< 10	Mg	0.5
N ₂	< 15	Mn	N < 0.5
C	6-10	Mo	N < 100
H ₂	2-4	Na	N < 20
F	5-10	Ni	N < 1
Ag	N < 0.5 ^a	Pb	N < 4
Al	N < 1	Rb	N < 2
As	N < 20	Sb	N < 6
B	N < 4	Si	< 2
Ba	N < 2	Sr	N < 20
Ca	N < 10	Te	N < 200
Cd	N < 8	Th	N < 80
Co	N < 1	Ti	N < 6
Cu	N < 0.5	Tl	N < 8
Fe	< 1	V	N < 8
Hg	N < 8	Zn	N < 20
In	M < 20	Zr	N < 20

^aN = not detected. In these cases the lower limit of detection by conventional spectrographic techniques is indicated.

Mechanical Properties

Hardness. Hardness as low as 250 DPH has been obtained.

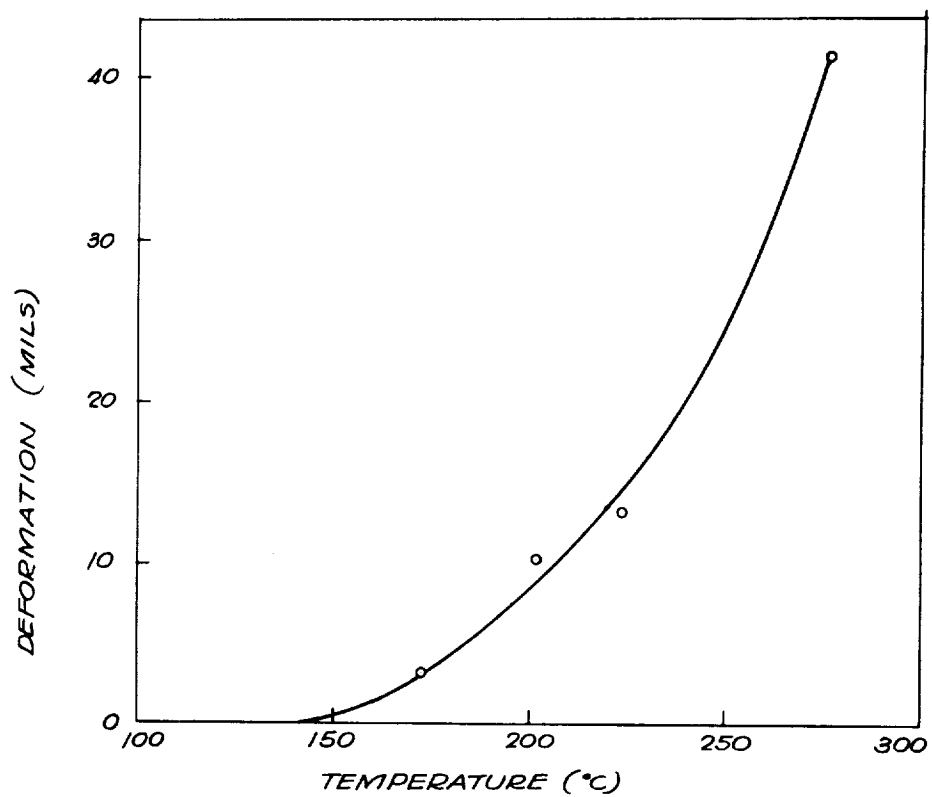
Ductility. Vapor deposition does not provide tungsten which is ductile at room temperature. However, a useful property to measure is the temperature at which the material undergoes a transition from brittle to ductile behavior. The temperature at which this transition takes place has been measured by ring compression tests (as shown in Fig. 4) and found to be about 150°C. This temperature is not significantly different from that exhibited by high-quality wrought material, and does not appear to worsen with heat treatment to 2000°C but may, in fact, improve slightly.⁽¹⁾ In wrought material, on the other hand, the ductile-to-brittle transition temperature increases by 300° to 500°C after recrystallization.



M-3034

(5 ×)

(a) Deformed vapor-deposited tungsten tube sections



(b) Plastic deformation as a function of temperature

Fig. 4--Ductile-to-brittle transition of vapor-deposited tungsten

Toughness and Reliability. Toughness, or the resistance to crack propagation, is difficult to measure, and no quantitative tests have been conducted with vapor-deposited tungsten; however, qualitative observations indicated that it is quite easy to handle and machine at room temperature. Very little unintentional breakage has occurred in the manufacture and handling of over 200 hardware items made by this process. No service failure of any vapor-deposited tungsten part has ever been observed at this Laboratory in tests both with and without nuclear environments and at temperatures up to 1800°C for over 3000 hr.

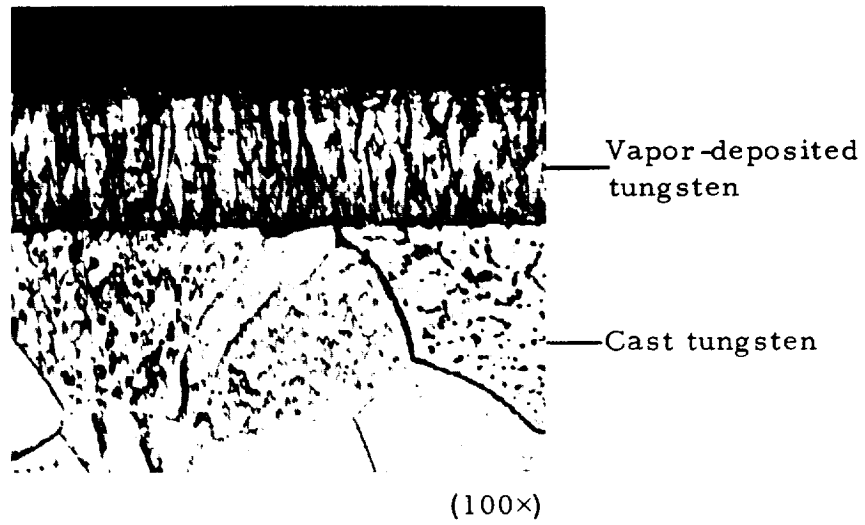
Strength. No room-temperature strength data are available except those derived from the ring compression tests, in which it was observed that stresses somewhat greater than 100,000 psi were required to cause fracture. High-temperature data (1370° to 2980°C) were obtained by the Jet Propulsion Laboratory⁽²⁾ and data at 2200°C were obtained by Solar Division, International Harvester Corporation.⁽³⁾ Both studies showed that vapor-deposited tungsten has high-temperature properties quite similar to those of wrought material. However, it was noted in Ref. 2 that this was only true if the material was not annealed prior to testing in a temperature range where void formation and grain growth occurred. This temperature was between 2200° and 2845°C for the material studied, which was supplied by High Temperature Materials, Inc. At General Atomic, vapor-deposited tungsten supplied by San Fernando Laboratories has been heated at 2500°C for 2 hr with no evidence of void formation.

Resistance to Grain Growth

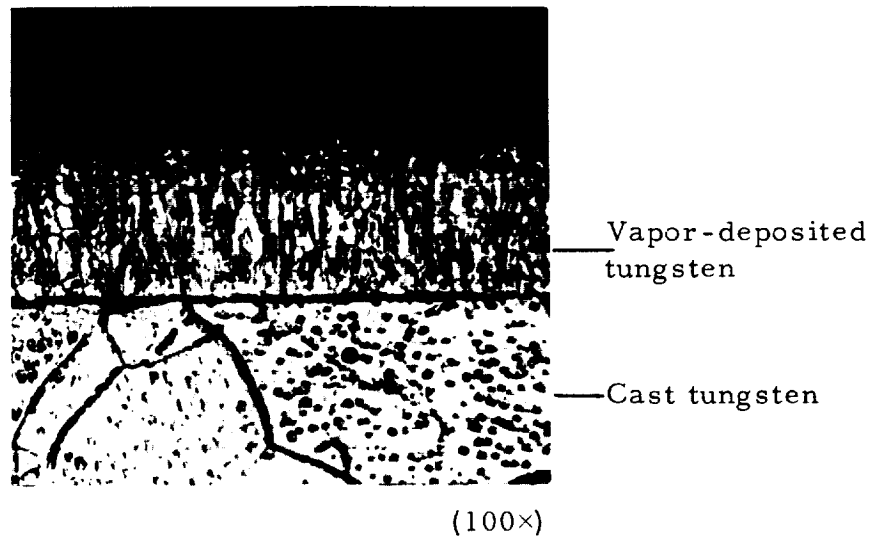
One of the major advantages of vapor-deposited tungsten is its ability to resist grain growth. Figure 5 illustrates the absence of grain growth in this material, even after 1100 hr at 1800°C ; Fig. 6 shows a series of pictures after various heat treatments as severe as 1 hr at 2500°C . Little, if any, grain growth was observable.

This resistance to grain growth appears not to be related to impurity contents and segregates in the metals, but rather to the lack of a driving force for grain growth. That is, the metal is highly oriented and has only low-angle grain boundaries, which eliminate surface energies at the grain boundaries as a driving force; the metal has little, if any, stored energy due to mechanical strain. If either of these conditions is altered, i. e., if high-angle grain boundaries or mechanical work is introduced, grain growth does occur.

The lack of correlation between grain growth and the metal chemistry is shown in Figs. 6 and 7 and recorded in Table 3, which shows the similarity in the composition of the specimens. The compositions differ significantly only in the oxygen content, with the specimen illustrated in

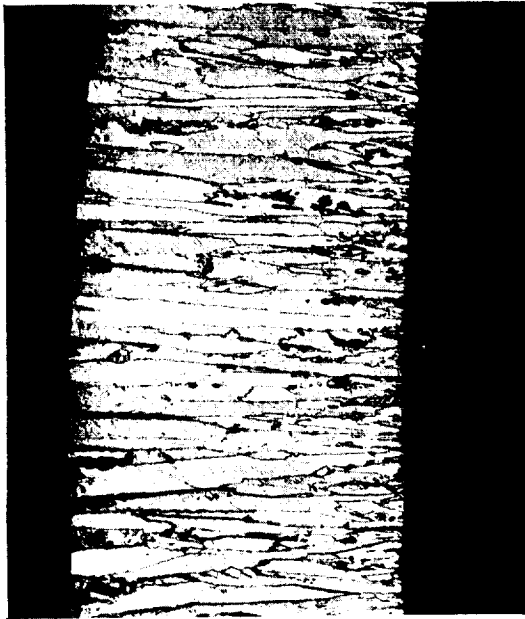


(a) As-deposited



(b) After 1100 hr at 1800°C

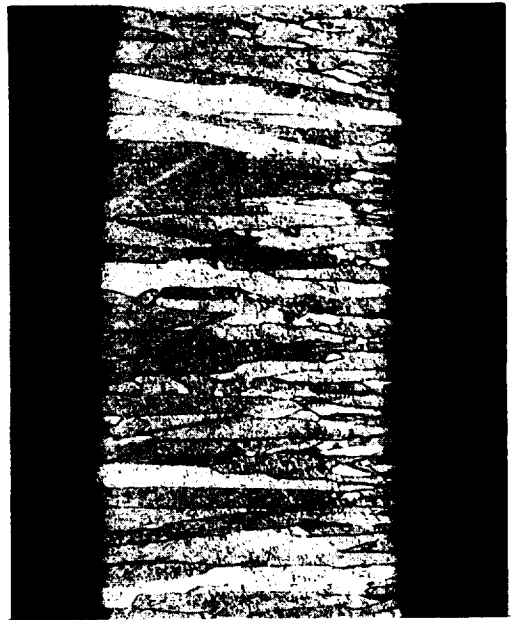
Fig. 5--Absence of grain growth in vapor-deposited tungsten after heat treatment at 1800°C (cast tungsten did not grow because grains were already extremely large)



M-8176-3

(100×)

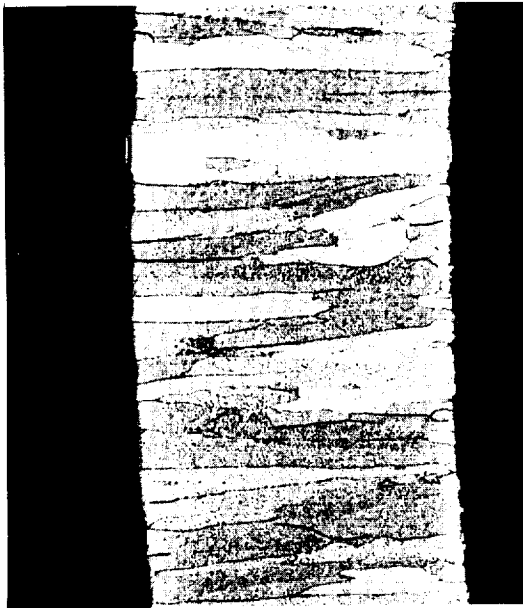
(a) As-received



M-8249-1

(100×)

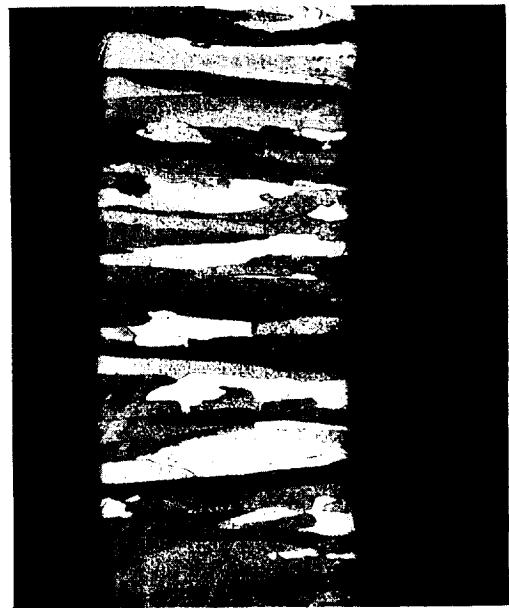
(b) 100 hr at 1800°C



M-8537-1

(100×)

(c) 15 hr at 2200°C



M-8584-1

(100×)

(d) 1 hr at 2500°C

Fig. 6--Structure of vapor-deposited tungsten tubing after high-temperature heat treatments (note lack of grain growth)

Fig. 7 having the higher impurity content. Yet the specimen shown in Fig. 7 has the maximum grain growth permissible, i. e., single grains going through the whole thickness of the tube wall. The difference between the two tubes was in the mechanical aspects of deposition; the tube shown in Fig. 6 was deposited over a male mandrel and that shown in Fig. 7 was deposited inside a female mandrel. This led to a difference in structure, in particular the volume and type of grains having high-angle grain boundaries. In Fig. 7a note the layer of tiny, randomly oriented grains on the outside diameter of the tube (where deposition began) and observe the manner in which growth initiates at these grains, as shown in Fig. 7b. The importance of randomly oriented grains in grain growth is illustrated in Figs. 8a and 8b.

Table 3

COMPARATIVE CHEMISTRY OF SPECIMENS ILLUSTRATING
LACK OF CORRELATION BETWEEN
CHEMISTRY AND GRAIN GROWTH

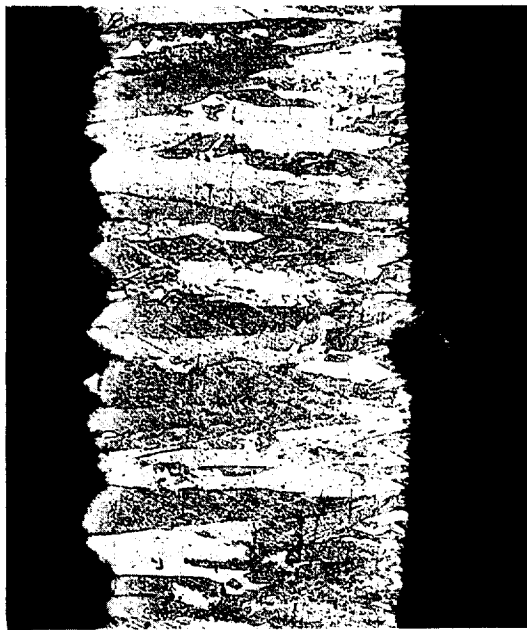
Element	Concentration (ppm) ^a	Concentration (ppm) ^b
Carbon	6.2	6.4
Oxygen	6.5	11.5
Nitrogen	5	4
Hydrogen	3	4
Fluorine	9	4
Iron	<1	<1
Magnesium	0.5	0.5
Nickel	N <1	<1
Silicon	<2	2

NOTE: No other metallic impurities were detected in either specimen by spectrographic analysis.

^a Specimen shown in Fig. 6.

^b Specimen shown in Fig. 7.

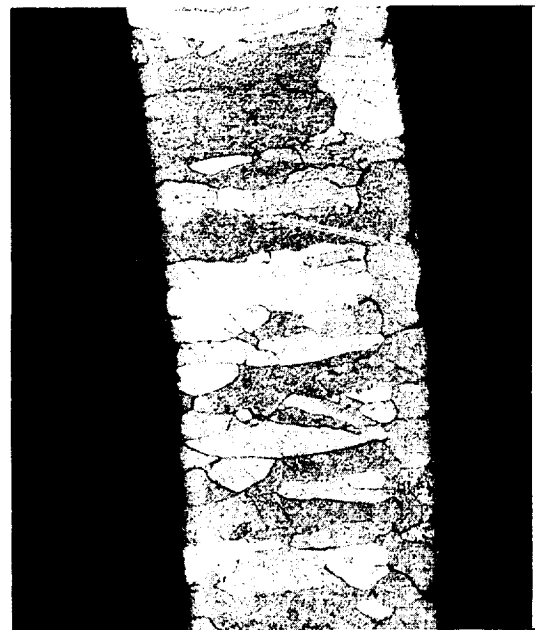
To obtain a relationship between grain-boundary angle and grain growth, an odd-shaped mandrel was prepared which had controlled "hills and valleys" to give a variety of grain-boundary angles, as the deposit accommodated to the substrate surface. Tungsten was deposited on this mandrel in the normal fashion, and rings were cut and thermally treated. Figures 9a and 9b show such sections in the as-deposited condition and after heat treatment at 2500°C. No grain growth is evident at any of the discontinuities, indicating that a greater degree of misorientation than simple tilt boundaries is required to initiate growth.



M-7778-2

(100×)

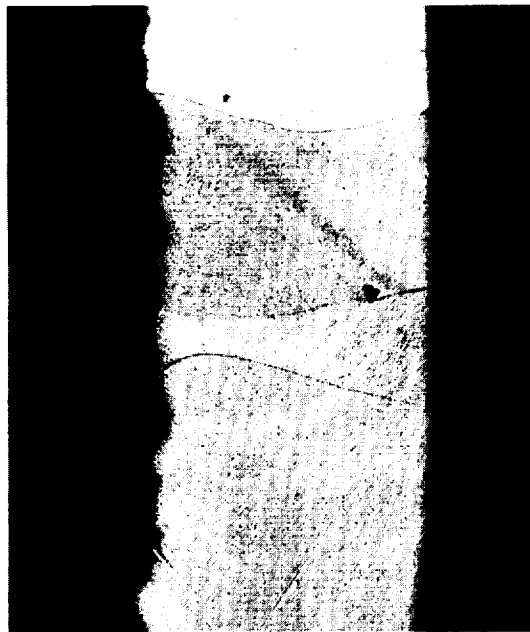
(a) As-received; note region of small misoriented grains on tubing OD



M-8250-1

(100×)

(b) 100 hr at 1800°C; note grain growth originating at tubing OD

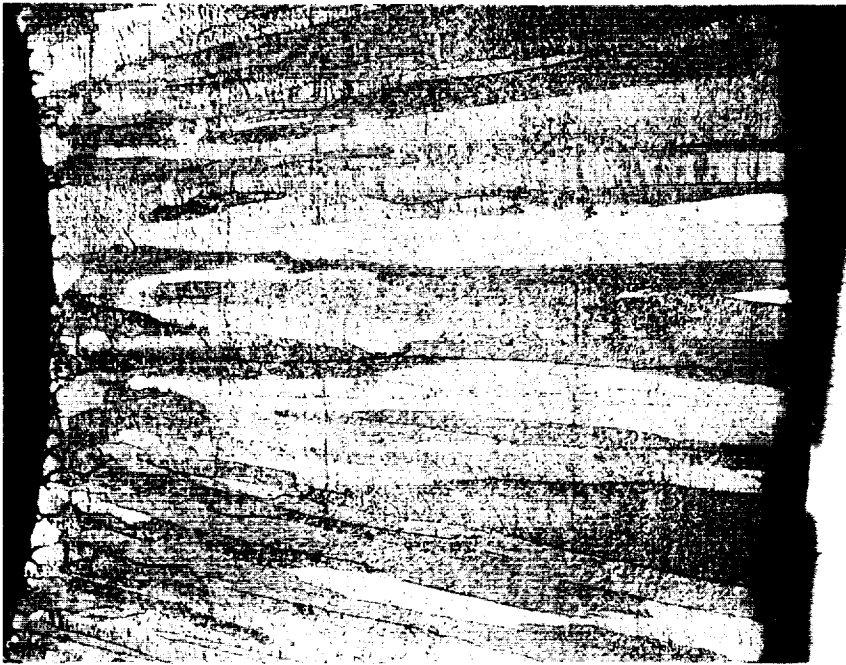


M-8585-1

(100×)

(c) 1 hr at 2500°C; note single grains penetrating through entire wall thickness

Fig. 7--Structure of vapor-deposited tungsten tubing formed in female mandrel after high-temperature heat treatments (note extensive grain growth)



M-8494-1

(100×)

(a) 15 hr at 2000°C; note randomly oriented, equiaxed grains at tubing ID



M-8599-1

(100×)

(b) 1 hr at 2500°C; note growth of equiaxed grains at tubing ID and manner in which they consume columnar grains

Fig. 8--Photographs showing grain growth initiating at randomly oriented grains formed at interface with substrate



M-8464-1-1

(a) As-received; note grain structures around discontinuities on tubing ID, wall thickness 0.070 in.



M-8603-1-1

(b) After heat treatment for 1 hr at 2500°C; note no observable change in grain structure showing that simple tilt boundaries are not sufficient to initiate grain growth

Fig. 9--Tungsten deposited on mandrel shaped to give a variety of grain structures

In those cases where growth does occur, stable interruptions formed between successive deposition layers can be effective in preventing single grains from penetrating through the entire wall thickness. This is illustrated in Fig. 10.

Having gained some understanding as to the mechanics of grain growth in this material, as described above, it has been possible to fabricate several hundred items of hardware which have successfully resisted any degradation due to grain growth during operational service.

Preferred Orientation

Highly oriented structures result from the deposition technique. The most common orientation is the $\{100\}$ plane parallel to the substrate surface; other orientations have been reported.⁽⁴⁾ In Fig. 11, which was taken by electron-emission microscopy, the patchiness of emission from a randomly oriented cast-tungsten specimen is compared with the uniform electron emission from a vapor-deposited specimen. This uniformity of work function is reflected in more of the surface of a thermionic emitter participating in the emission, thereby increasing the performance.

Machining

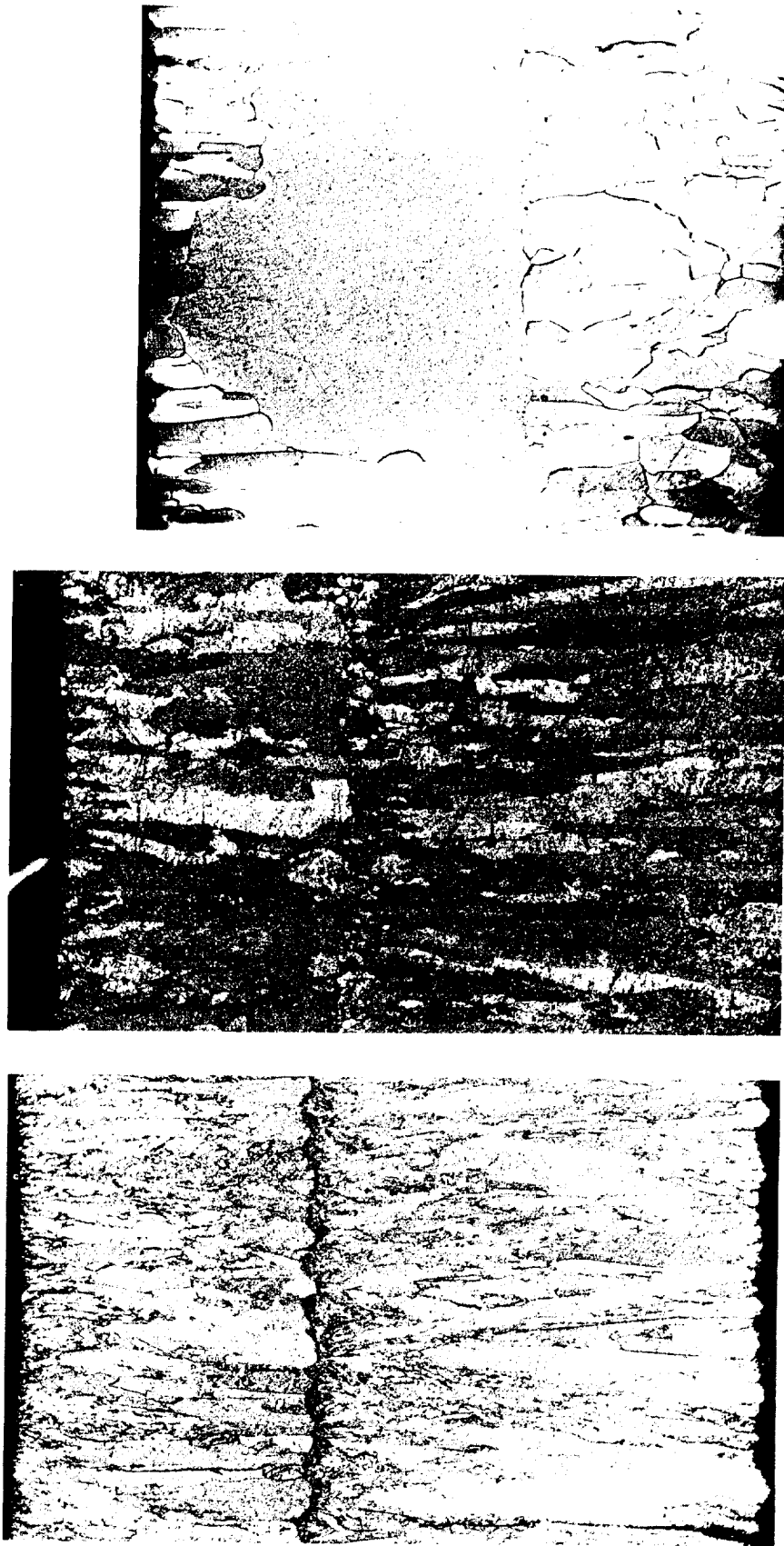
Vapor-deposited tungsten is easier to machine than either cast or pressed and sintered material because of its smaller grain size and greater toughness. Most machining is done by grinding and electrical discharge techniques. However, holes are frequently drilled with carbide drills and some turning has been done with high-speed steel tools.

Joining

Complex shapes of vapor-deposited tungsten can often be made without the need for joints (e. g., the irradiation specimen described below), but it is still sometimes necessary to use joints when producing certain shapes.

Since vapor-deposited tungsten is used because of its unique grain structure, welding is not a desirable form of joining because it destroys the characteristic grains to form a cast structure. Diffusion bonding is readily performed, as illustrated in Fig. 12. Diffusion bonding conventional tungsten is difficult because the bonding temperature must be restricted to below the recrystallization temperature to avoid embrittlement. Since the vapor-deposited tungsten does not show grain growth or recrystallization, higher bonding temperatures can be used and better bonds are obtained.

In addition to diffusion bonding, the vapor-deposited tungsten can be readily brazed. Vanadium, copper-titanium, and nickel base brazing alloys have all been employed successfully.



M-4570-1-1

(70×)

(a) As-received; interruption in columnar growth formed by stop-ping plating process and grinding surface with bolt sander

M-4570-2-1

(70×)

(b) After 95 hr at 1800°C; note small equiaxed grains at interruption and ID surface

M-4792-1-1

(70×)

(c) After 2500°C for 1 hr; few very large grains formed but do not cross interruption, large grains appear to be growing sideways within individual layer

Fig. 10--Effectiveness of interrupted deposition in resisting grain growth

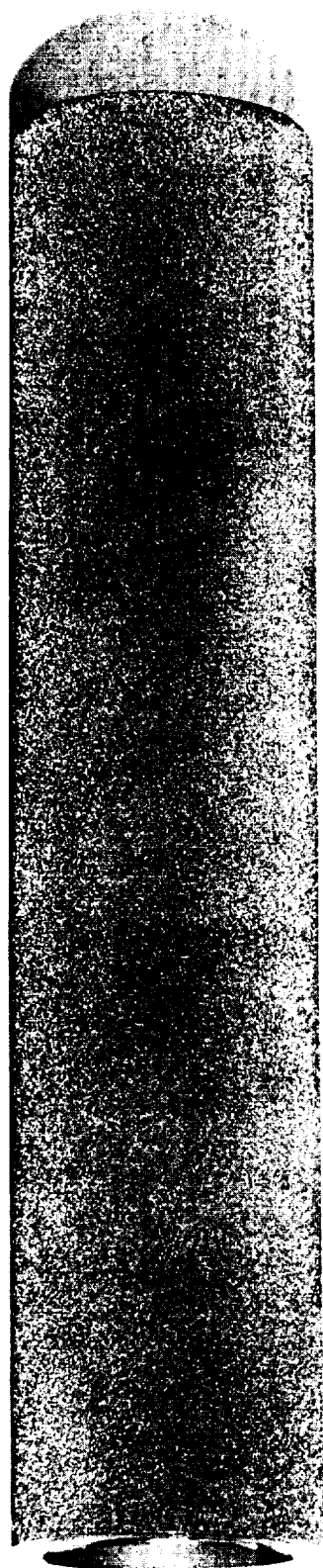


(a) Emission pattern of cast tungsten



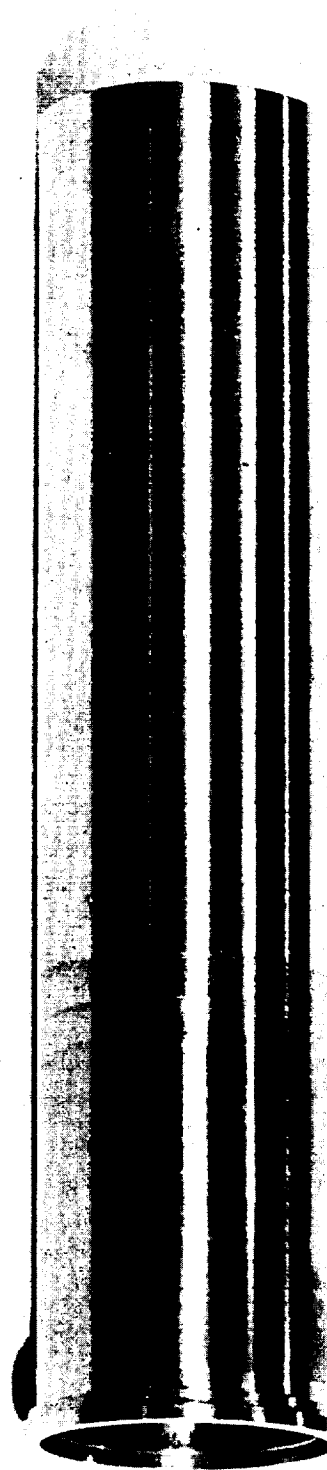
(b) Emission pattern of vapor-deposited tungsten over UC

Fig. 11--Electron-emission micrographs of cast and vapor-deposited tungsten showing improvement in uniformity of emission in vapor-deposited specimen



TE-22348

(a)



TE-22350

(b)

Fig. 12--Vapor-deposited tungsten thermionic emitter incorporating a tungsten-to-tantalum diffusion bond: (a) tungsten blank; (b) machined emitter after bonding to tantalum stem

FABRICATION OF HARDWARE ITEMS

As previously mentioned, quite complex shapes can be fabricated by this technique. Several of the more than 200 pieces of hardware already made by this process are shown in Figs. 12 through 15. Figure 12b shows an emitter formed by diffusion bonding the closed-end tungsten blank shown in Fig. 12a to a tantalum stem and machining to size.

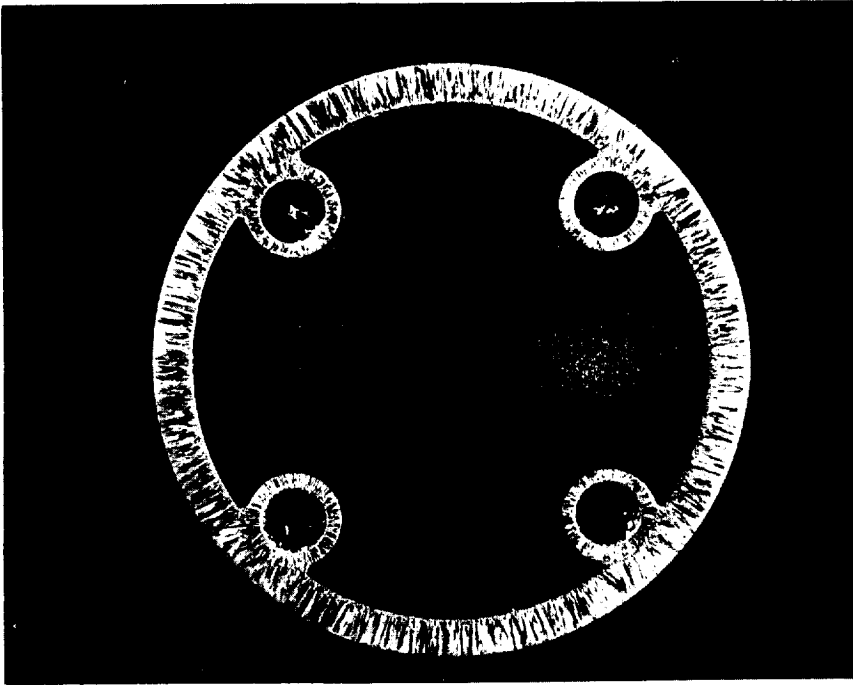
Figure 13 shows a modification of the basic emitter structure illustrated in Fig. 12, in which uranium carbide-zirconium carbide fuel slabs were embedded in the tungsten emitter blank during deposition. Figure 14 shows another modification in which four thermocouple-protection tubes were buried in the wall of the emitter during fabrication.

One of the most complex shapes prepared was the irradiation specimen shown in Fig. 15. This was especially difficult, since a void had to be included in the shape.

These are only samples of what has been done at General Atomic and illustrate the flexibility of the vapor-deposition process.

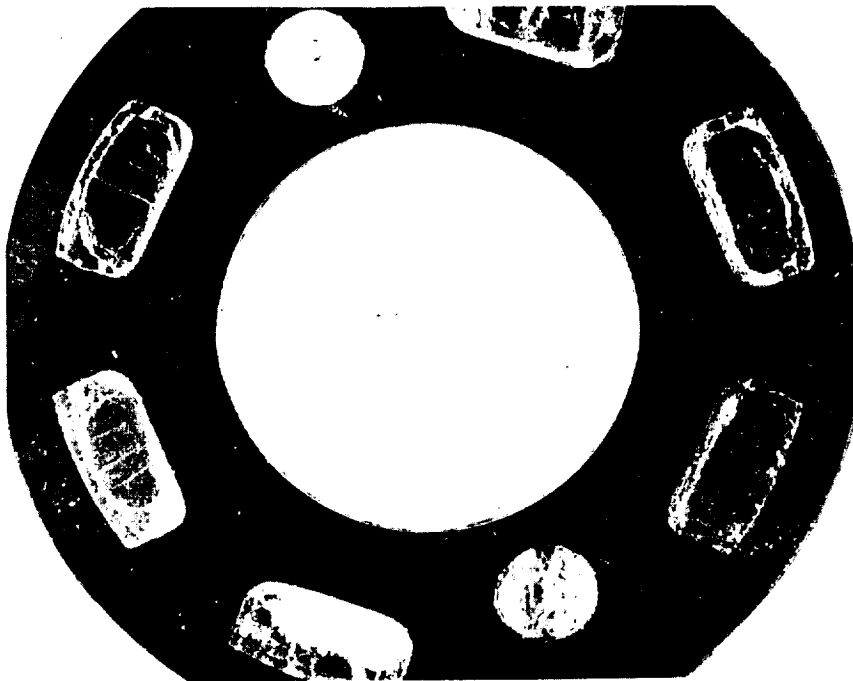
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M-4591

Fig. 14--Cross section of modified emitter in which four thermocouple wells were embedded in emitter wall during deposition



M-6424-1-1

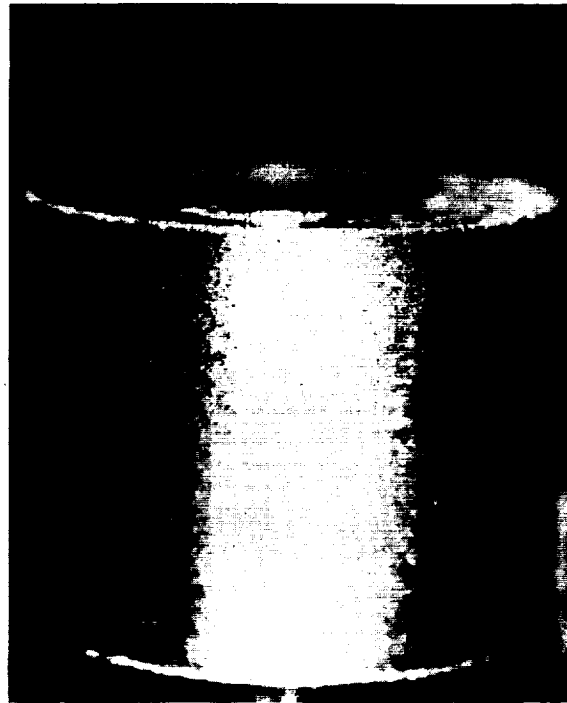
Fig. 13--Cross section of modified emitter in which UC-ZrC fuel slabs were embedded during deposition



W-UO₂ cermet clad with vapor-deposited tungsten



Top View



Side View

90 mol-% UC-10 mol-% ZrC (depleted) clad with vapor-deposited tungsten

Fig. 15--Irradiation specimen; void volume was formed to allow space for fission gases released during irradiation

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